

**United States Department of Agriculture
Agricultural Marketing Service, Science & Technology
Pesticide Data Program**

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| SOP No.: PDP-QC-02 | | Page 1 of 6 |
| Title: Initial Laboratory Evaluation | | |
| Revision: 6 | Replaces: 8/1/96 | Effective: 10/1/98 |

1. Purpose:

To provide USDA/AMS Pesticide Data Program (PDP) a mechanism by which to initially evaluate a laboratory or to evaluate entirely new methodology within a laboratory.

2. Scope:

This SOP shall be followed by all analytical laboratories which are conducting residue studies for PDP. This includes laboratories conducting stability and other studies which may impact the program.

3. Definitions:

4.

Refer to Glossary.

4. Outline of Procedure:

- 5.1 Evaluation Guidelines
- 5.2 Analytes/Commodities
- 5.3 Establishment of LODs and LOQs
- 5.4 Determination of Consistent Instrumental Response
- 5.5 Determination of Method Performance
- 5.6 Precision and Accuracy Data Collection
- 5.7 Method Evaluation Reporting

5. References:

Chemist Qualification document from Robert Epstein and summarized by Terry Jackson with comments from Participating State Laboratories, 4/23/92

Validation of Methods Used in the Florida Department of Agriculture and Consumer Services' Chemical Residue Laboratory, Parker, G.A., JAOAC, 74, No. 5, pp. 868-871, 1991

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GLP Meeting with EPA/OPP, EPA/OCM, USDA/AMS, and USDA/AMS GLP Committee, 4/28/92

GLP Meeting with USDA/AMS GLP Committee and Robert Epstein, 4/29/92

Evaluation of Analytical Methods Used for Regulation of Foods and Drugs, Horwitz, W., Analytical Chemistry, Vol. 54, No. 1, pp. 67A-76A., 1982

Quality Assurance Principles for Analytical Laboratories, Garfield, F., AOAC, 1991

Quality Assurance of Chemical Measurements, Taylor, J.T., Lewis Publishers, 1989

Letter, Martha Lamont to PDP participants, May 5, 1992

Quality Assurance Officer's Meeting, February 21-23, 1995

Quality Assurance Committee Conversations, March-May 1995

Laboratory Comments Generated from QC-2, Draft 05-30-95, July-September 1995

6. Specific Procedure:

6.1 Evaluation Guidelines

- a. This is an evaluation requirement for new PDP laboratories or those laboratories conducting new studies for which PDP requires a QC-2 evaluation.
- b. The methodology, method evaluation records, and team or individual qualification documentation shall be filed with the Quality Assurance Unit (QAU).

6.2 Analytes / Commodities

- a. The analytes subjected to the initial laboratory evaluation study shall include all marker pesticides as required by the PDP Technical Program Director or those specific to single analyte methods undertaken by a particular laboratory.
 - b. For laboratories performing multiresidue analysis, PDP shall provide a written list of commodity groups requiring method evaluation.
 - c. For laboratories performing single analyte analysis, PDP shall provide a
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written list of the analyte/commodity pairs requiring method evaluation.

6.3 Establishment of LODs and LOQs

See SOP QC-10, Determination of LODs and LOQs. The establishment of LODs and LOQs must be completed before proceeding to Section 6.4.

6.4 Determination of Consistent Instrumental Response

- a. Standards containing marker pesticides/single analyte analysis pesticides shall provide consistent responses at levels from LOQ to 10xLOQ on the primary injector and detector system (refer to glossary). If additional injector and detector combinations are to be used for quantification, they must be likewise evaluated.
- b. A Three Point Standard Curve from LOQ to 10xLOQ is the minimum requirement to show consistency of instrumental response (more can be done if desired).
- c. **Standards in Matrix:**
For each marker or single analyte/commodity pair (use "worst-case" commodity determined from QC-10) run each level three times over three days for a minimum of nine points.
- d. **Standards Not in Matrix:**
For each marker/single analyte run each level three times over three days for a minimum of nine points.
- e. Prepare summary form(s) of the acquired data (See Attachment QC-2.1).

6.5 Determination of Method Performance

- a. Fortified samples shall be run through the entire analytical method on the primary injector and detector system (refer to glossary). If additional injector and detector combinations are to be used for quantification, they must be
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likewise evaluated.

- b. Fortify samples in triplicate at approximately LOQ, 5xLOQ, and 10xLOQ for each marker pesticide in the “worst-case” commodity. Take these fortified samples through the entire analytical method (treat as a set of nine samples). A reagent blank and a matrix blank shall be subjected to the analytical method along with the fortified samples.
- c. For each data point, calculate the Percent Recovery compared to known standards to three significant figures if greater than 100% or to two significant figures if less than 100%. Standards prepared and run in Section 6.4 may be used.
- d. Calculate the Coefficient of Variation (%CV) for each analyte/commodity pair (refer to Glossary).
- e. Prepare summary form(s) of the acquired data (6.5c and d) by analyte, level and commodity group (See Attachment QC-2.1). Refer to QC-4 for PDP acceptance criteria.

6.6 Precision and Accuracy Data Collection

- a. The precision and accuracy data collection shall be compiled from the commodity groupings as specified by PDP. Each marker pesticide/single analyte pesticide shall be spiked at 2xLOQ and evaluated using a minimum of seven data points, with at least two points from each commodity in the group.
 - b. The required data points shall be obtained from matrix spikes concurrent with sample analysis.
 - c. For each data point, calculate the Percent Recovery compared to known standards to three significant figures if greater than 100% or to two significant figures if less than 100%.
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- d. Calculate the Coefficient of Variation (%CV) for each analyte/commodity pair (refer to Glossary).
- e. Prepare summary form(s) of the acquired data (See Attachment QC-2.1). Refer to QC-4 for PDP acceptance criteria.

6.7 Method Evaluation Reporting

- a. The methodology, method evaluation records, summary form(s), and any other raw data generated during QC-2 evaluation shall be maintained by the QAU.
 - b. Once the laboratory has completed Sections 6.3 - 6.6 of QC-2, the summary form(s) are sent to the PDP Technical Program Director.
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|---|--|
| Attachment QC-2.1b, 10/01/98 | |
| <i>Determination of Method Performance</i> | |
| USDA, AMS - Pesticide Data Program | |
| Commodity: | |
| Date: | |
| Lab: | |

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| Attachment QC-2.1b, 10/01/98 | |
| <i>Determination of Method Performance</i> | |
| USDA, AMS - Pesticide Data Program | |
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| Attachment QC-2.1b, 10/01/98 | |
| <i>Determination of Method Performance</i> | |
| USDA, AMS - Pesticide Data Program | |
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| Attachment QC-2.1c, 10/01/98 | |
| <i>Precision and Accuracy Data Collection</i> | |
| USDA, AMS - Pesticide Data Program | |
| Commodity: | |
| Date: | |
| Lab: | |

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| Attachment QC-2.1c, 10/01/98 | |
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| USDA, AMS - Pesticide Data Program | |
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| Attachment QC-2.1c, 10/01/98 | |
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| Date: | |
| Lab: | |

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